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BUTYL INSULATION

BY

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ESSO LABORATORIES
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Butyl Insulation

by

W. C. Smith

I Introduction

Butyl rubber has been of considerable interest to the wire and cable industry ever since the early 1940's. This is because it is inherently resistant to ozone, heat aging, has excellent electrical properties, and is vulcanizable. The recently announced advances in heat, moisture, and ozone resistance, coupled with the good availability outlook, have greatly stimulated the demand for Butyl.

It is the purpose of this paper to present information on Butyl rubber for the wire and cable industry, to provide facts and observations which will serve as a base for development of sound compounds for the industry's varied interests.

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Information has been grouped under properties desired in insulation: processing to give smooth non-swollen extrusions, ozone resistance and ways to obtain the optimum in resistance to cracking, heat aging, electrical properties and their stability in water, dielectric strength, a comparison of sulfur with quinoid cures, and a section on jacketing.

II Processing

A. Fillers

The general processing of Butyl has been covered in another publication (1). However, a number of specific fillers which appear to have particular merit for insulation in at least one of their properties are listed in Table I.

In ability to deaden the stock and promote smooth extrusions, in order of effectiveness, are Zeolox 20 (Na Zeolite), Alon S (Al_2O_3), all diatomaceous earths represented by Dicalite SA-3, fine particles whiting such as Purecal U, and high calcined clays, Whitetex and Burgess Iceberg.

Deadening is not necessarily accompanied by fast extrusion due to filler structure formation in the stock. Rated by speed of extrusion, diatomaceous earths are best, followed by high calcined clays, and Purecal U. Alon S, to a high degree, and Zeolox, to a lesser extent, produce stocks which stick to all metallic surfaces. This deficiency of Alon S has not been overcome, and of Zeolox only impractically.

The wide range of tensile and modulus characteristics offered by these fillers and Cab-O-Sil provides considerable latitude in the formulation of insulation compounds.

In original electrical properties, with the possible exception of the Zeolex compound*, values range from excellent to fair. The high calcined clays appear to be a reasonable compromise in all properties.

B. Plasticizers

Plasticizers in Butyl almost invariably increase the swelling without greatly increasing the extrusion rate. Some smoothing of the extrusion is usually noted although the chief contribution lies in softening the stock and reducing scorch. In this category are included the process oils, waxes, petrolatum, stearates, mineral rubber, Cumars and other similar resins. Factices severely retard the cure of Butyl and are not exceptional in water stability. Up to 25 parts on the gum definitely increases extrusion rate, smoothness and reduces swell, and tendency to sag on wire. Resins such as Marbon and Pliolite are cure retardants, and aside from a smoothing action do not improve extrusion.

Plastics such as polyethylene and Parapol S are inert to cure. They smooth extrusions and increase modulus and shore.

C. Stiffeners

Often where pan curing is employed, Butyl compounds tend to sag on the wire. This can be overcome in several ways. The use of

* Compound contains tributoxyethyl phosphate for mold release.

stiffening plastics such as polyethylene and Parapol 8 helps to some degree, as does the use of higher Mooney Butyls. Even more effective is a Polyac treat. Here the Polyac, 0.1 to 0.4 based on the polymer, is first dispersed cold in the rubber, then alone or with fillers is subjected to dynamic mixing (mill or Banbury) for five minutes at 300°F. or its equivalent. This amount of heating creates maximum cross linking with resultant decrease in plasticity and increase in nerve. Additional treatment at high temperatures serves to decrease nerve (smoother extrusion) and further decrease plasticity. Another method for stiffening the compound is the addition of a filler such as Cab-O-Sil. The following results were obtained in a standard (see Table IV) power cable formula:

| | <u>Williams Plasticity - Recovery 5 Kg at 70°C.</u> |
|-----------------------------|---|
| Control | 129-13 |
| +10 Kenflex A | 122- 7 |
| +10 Kenflex A +5 Cab-O-Sil | 126-10 |
| +10 Kenflex A +10 Cab-O-Sil | 138- 6 |

When Kenflex A is used for improved ozone resistance, as will be shown later, a softening effect results. This softening can be balanced by five parts of Cab-O-Sil, while 10 parts greatly stiffen the compound.

D. Mixing Technique

Compounds can be satisfactorily mixed on the mill or in the Banbury in conventional cycles and temperatures; however, much improvement is realized if compounds can be mixed hot. The gain is realized in subsequent processing, physical properties, dielectric strength and in stabilizing heat aging.

Three types of mixing have been employed in this work, the pertinent type being indicated in each table.

Normal mixing involved simply the addition of compounding ingredients to Butyl on a cold (100-160°F.), 6" x 12" laboratory mill. Zinc oxide and stearic acid were added first, followed by filler, waxes, oils, etc., resins added to a small aliquot of polymer and blended in, finally accelerators and vulcanizing agents and completed by conventional ASTM mixing.

Special mixing involved the hot milling (6" x 12" laboratory mill) for five minutes at 300°F. of Butyl alone or with any high melting resin, followed by the cold mill addition of all compounding ingredients mixed together. The mix was completed by ASTM blending and four pass refining at 0.010 inch mill setting.

Hot Banbury mixing involved charging all ingredients, except accelerators, in a conventional cycle to a Banbury at 280°F. Temperature was allowed to build up to 325-360°F. Accelerators were added on a cold

mill. Improvements in physical properties and heat aging resulting from hot milling can be observed by comparing the GR-I-35 control stocks (without Kenflex A) in Tables X and XI. The effect of hot milling on dielectric strength is found by making a similar comparison of Table VIII and Tables VII, IX, and XVI.

The advantages of hot Banburying over normal milling are shown by the processing and physical property data in Table II and by the dielectric strength data in Table XIV. In addition to these effects there is a marked reduction in the porosity of cures as determined either on press or steam cured samples.

The dispersion of zinc oxide has presented a problem in thin gauge extrusion of mechanical goods. The oxide and some undispersed filler are forced out of the stock and build up on the die. Thus it is likely that the dispersion is also insufficient in insulation compounds. In mechanical goods the problem was mitigated by premixing the zinc oxide powder with filler, by upside down Banbury mixing, or by hot Banburying (350°F.). Such procedures have been rewardingly followed in the special mix and hot Banbury mix outlined in this paper.

III Properties of Quinoid Cures

A. Ozone Resistance

In recent years as the wire industry has geared to higher voltage and temperature insulation, demands for greater ozone resistance have been imposed. Butyl rubber has always been outstanding in this regard and no great difficulty was experienced in meeting the pertinent ASTM D-574 specifications. With the increased requirements for ozone resistance, it has become necessary to analyze more comprehensively the basic resistance of commercial Butyl polymers to ozone attack and the effect on these of compounding ingredients in the several types of cures available. It was virtually impossible to screen these many potential variables under actual service conditions and became necessary to resort to the common practice of accelerated tests. As the level of ozone resistance was improved, time considerations made it necessary to increase the severity of the test.

Testing Procedure

Ozone was generated and determined according to ASTM D470-49T with a few minor exceptions in the drying train and test chamber.

Several test conditions were employed in this work. The specific conditions will be noted in each table and figure. Vulcanizates were

prepared by press molding. Pads were 0.075 ± 0.005 or 0.018 ± 0.002 inch in thickness. Samples were die-cut in the form of square shoulder T-50 specimens, body width $1/12$ inch, and length one inch. Prior to placing the sample in the extension apparatus, the square shoulders were coated with silicone grease to minimize cracking at these points of excessive stress. After elongation of 50 per cent, 30 minutes relaxation time was allowed to elapse before exposure to ozone. Ozone concentration was run at two levels, 0.012 and 0.2 per cent by volume. Since it is quite difficult verbally to transmit an impression of degree of cracking, time to breakage was selected as a measure of ozone resistance.

The variable adjustment-extension apparatus pictured in Figure 1 is an improved model permitting tapered-necked 0.1" wide T-50 dumbbells to be used and overcomes the problem of jaw breaking. No silicon grease is needed. Tests employing this dumbbell were run at 100% elongation in 0.2 vol. % ozone.

1. Effect of Unsaturation

Butyl rubber is produced commercially in the government plants under the GR-I designation. Currently the following grades are made:

| <u>Grade:</u> | <u>Approximate Unsaturation (2)</u> | | <u>Mooney</u> |
|---------------|-------------------------------------|-------|---------------|
| | <u>Range</u> | | |
| GR-I-35 | 0.6 - 1.0 mole per cent | | 38-47 |
| GR-I-50 | 1.0 - 1.5 | " " " | 41-49 |
| GR-I-15 | 1.5 - 1.9 | " " " | 41-49 |
| GR-I-17 | 1.5 - 1.9 | " " " | 61-70 |
| GR-I-18 | 1.5 - 1.9 | " " " | 71+ |
| GR-I-25 | 1.9 - 2.3 | " " " | 41-49 |

The unsaturation of all the above polymers is extremely low compared to natural rubber at 100 mole per cent. Within this unsaturation range of 0.6 to 2.3 mole per cent, however, there is an extraordinary difference in resistance to ozone, the greatest change occurring in the GR-I-35 range. This is demonstrated in Figure 2. Possible deviation within the GR-I-35 specification explains variable ozone resistance both in the present work and in the field.

2. Effect of Compounding Ingredients

a. Softeners, Plasticizers

Paraffin wax and stearic acid have been shown to be beneficial (1) to ozone resistance. Process oils are detrimental (Table III) and ester plasticizers are highly detrimental. It is felt that these materials, in relation to their solubilizing action, open up the structure and in the case of esters increase ozone solubility (3), allowing for deeper surface penetration of ozone. It is known* that both classes of plasticizers reduce Butyl rubber resistance to permeation of gases.

* Unpublished data.

b. Resins and Polymers

Table IV shows the results of compounding GR-I-35 with various resins and polymers. Most of the resins tested are relatively ineffective and in view of this, the remarkable resistance to ozone imparted by Kenflex A is astounding. This resin is reported to be a condensate primarily of dimethyl naphthalene and formaldehyde and thus, structurally, does not differ profoundly from most of the other resins tested. It will be noted that the compound containing 20 parts of Kenflex A showed no cracking whatsoever after 50 hours of exposure at 0.2 volume per cent ozone at 50% elongation and resisted breaking over 8 hours at 100% elongation. Kenflex A extends its protective power to a marked, but lessening degree, as the unsaturation of Butyl is increased (Figure 2). Ten parts of Kenflex A confer to GR-I-25 the basic ozone resistance of GR-I-50, and to GR-I-15 the ozone resistance of GR-I-35. Other properties of the resin will be found in this paper under heat aging, electrical stability, and dielectric strength. Polymers such as Parapol-S (formerly S-Polymer) and Vistanex B100 contribute to ozone resistance (Table IV), the latter strongly and to a considerably greater extent than would be anticipated by dilution of the unsaturation.

c. Fillers

An extensive loading study with respect to ozone resistance has not yet been undertaken. Spot information shows that ozone resistance

decreases with loading and that clays and hydrated alumina are superior to natural whiting (Table V).

3. Cure State

Buckley and Robison have shown that ozone resistance increases appreciably with state of cure in sulfur vulcanized stocks (3). An explanation of this effect is the hindrance of the double bond by formation of a cross-link on the alpha carbon. This trend is reversed strikingly at the point where reversion sets in. This is understandable from the viewpoint of severance of the cross-links of vulcanization and the resulting removal of hindrance around the double bonds involved. Weakly increasing ozone resistance with vulcanization appears in quinoid cures (Table VI) in high unsaturated Butyl. Since this type cure yields a plateau and reversion is not experienced over prolonged cure times, no degradation of ozone resistance has been found.

The so-called Polyac treat of Butyl probably falls into this same category. In this treatment small amounts of Polyac are cold milled into Butyl, the mass is then hot milled. This effectively cross-links the rubber and does so far more efficiently than a static heating of the same duration. Table VII shows the resultant marked increase in ozone resistance.

4. Various Quinoid Cure Types

There are a variety of combinations of curatives possible within the quinoid type category. Five of these, and their counterparts with Kenflex A, are shown with their general properties in Table VIII. One of these combinations contains no sulfur, for applications where minimum copper corrosion is desired. Under the conditions of ozone testing - 0.2 volume per cent ozone and 50 per cent elongation - there is considerable variance in resistance ranging between 170 and 1550 minutes on the controls. These figures compare with 510 minutes on the standard Dibenzo GMF-Pb₃O₄ formula. In all but one instance Kenflex A increases ozone resistance to over 3000 minutes. There is no visible ozone attack. A test at 100% elongation permits destructive evaluation of the stocks with Kenflex A. The Dibenzo GMF-Pb₃O₄ cure appears the best, requiring 300 minutes to break.

5. Heat Aging Effect

If the postulation of a correlation between cross-links and ozone resistance is correct and we reaffirm the resistance of quinoid cures to reversion, then the combined effects of this and oxidation in heat aging of these stocks should only serve to increase ozone resistance. This is borne out by heat aging at 300°F. for as long as 72 hours (Table IX). The tremendous increase in ozone resistance at 72 hours at 300°F.

is perhaps misleading since at this level of aging, samples are flabby and develop low stress at 50 per cent elongation. Degradation here may be due to chain rather than cross-link severance.

B. Heat Aging

The heat resistance of Butyl quinoid vulcanizates is such that the conventional accelerated aging tests employed on natural rubber result in no deterioration. It is necessary to age at 250°F. or 300°F. to obtain significant changes under reasonable time considerations. In the present work 300°F. was employed. The standard formulation referred to in the tables was as follows:

| | |
|--------------------------------|-----|
| GR-I-35 | 100 |
| Zinc Oxide | 5 |
| Stearic Acid | 1.5 |
| Whitetex #2 | 90 |
| Dibenzo GMF | 6 |
| Pb ₃ O ₄ | 9 |
| Sulfur | 1.5 |

1. Effect of Unsaturation

As determined in the standard formula, specially mixed, the effect of increasing Butyl unsaturation (Table X) on the tensile strength and modulus retained is negligible, and on elongation retained is fairly constant, at unsaturations above GR-I-35. Kenflex A provides a gain in per cent retention of all three properties but it must be noted that this figure is strongly influenced by the level of the unaged physicals.

2. Effect of Additives

A GR-I-35 control stock identical to the above but normally mixed (Table XI) produces considerably lower physical properties and yields less resistance to 300°F. aging. However, within this normally

mixed series, increasing use of Kenflex A improves aging. Both original and aged tensile strength are benefited by the further addition of Cab-O-Sil. In view of other work, it is felt that the main Cab-O-Sil contribution here is a stabilizing effect much as a special mix stabilizes aging results compared to normal mixing.

Parapol S and Vistanex B-100 in this formulation contribute little or nothing to heat aging.

A complete Cab-O-Sil loading to the extent of 25 volumes (Table XII) in a quinoid cure provides amazingly good physical properties, retained well in heat aging. Since it is an anhydrous silica, it yields good electrical properties and electrical stability in water. The deficiencies lie in the stiffness of these stocks and the currently high price. It can readily be seen why the addition of small amounts of this filler can upgrade the properties of the standard formula.

3. Effect of Cure Type

There are a number of variations possible within the quinoid cure, some of which are shown in Table VIII, both with and without Kenflex A addition. Air heat aging of 3 days at 300°F. shows marked differences in resistance ranging from almost complete inertness to 23 per cent tensile retention. The data must be examined both in the light of the level of aged properties and the per cent retained. The best balance is obtained

with the GMF-Altax-Sulfur type of cure, yielding 670 psi aged tensile or 65 per cent retained and 61 per cent elongation retained. Consideration should be given to a GMF-Altax-Pb₃O₄ cure which has remarkable aging characteristics, although at a lower absolute level, and may prove to be outstanding under more severe aging tests. Comparing the control GR-I-35 stocks in Tables XI and VIII shows the non-reproducibility of aging obtained from the normal mix. A comparison of the stocks containing Kenflex - one special and one normal mix - shows the advantage gained in hot milling.

4. Inert Atmosphere

Aging in an inert atmosphere, in this case nitrogen (Table XIII), yielded, as would be expected, a marked increase in resistance. This situation is approached in rubber jacketed cables and probably reached in tightly sealed metal jacketed cables.

C. Electrical Properties and Stability in Water

The dielectric constant and δ power factor of raw pure gum Butyl are exceptionally good and remain good in a pure gum vulcanizate:

| Raw Butyl | IKC | Frequency | |
|-----------------------|-----------------|-----------|---------|
| | | 50MC | 1300 MC |
| Dielectric Constant | 2.3-2.35 | 2.2-2.3 | 2.12 |
| δ Power Factor | 0.05-0.09 | 0.03-0.09 | 0.04 |
| Cured Pure Gum Butyl | 2.4-2.6 0.11 | | |

In the early work with Butyl insulation compounds, electricals were run on press pads between unweighted electrodes. In subsequent work it was felt, due to difficulties in obtaining perfect pads, that weighted electrodes should be used. A study of this showed that a six kilogram weight provided optimum control without compressing the pad. Naturally the increased contact produced electrical values of a more accurate but higher order and this fact should be taken into account when comparing results in the various tables.

1. Effect of Fillers

Comparing a natural whiting with Whitetex clay (Table XIV), there is an improvement in the dielectric constant of the compound as successive portions of whiting are replaced with Whitetex. This effect is the same whether the compounds are normally milled or hot Banbury milled and the same relation holds both before and after water immersion. There is no conclusive effect on power factor although it appears best in mixed loadings.

Cab-O-Sil loadings (Table XII) show excellent original electrical properties which do not degrade excessively on water immersion at 85°C.

2. Effect of Additives

The effect of adding Kenflex A, Cab-O-Sil, and Vistanex B-100 on original electricals and the stability in water are shown in Table XV. Kenflex A appears to be slightly detrimental in water absorption and

electrical stability. However, this can be completely overcome and an improvement made in both regards by the addition of small amounts of Cab-O-Sil. It will be recalled that this same approach was used to overcome the softening effect of Kamflex A in the raw stock and to upgrade the physical properties.

The use of Vistanex B-100 reduces water absorption slightly but somewhat adversely affects the power factor stability.

3. Cure Type

A number of variations on the quinoid type cure (Table VIII) show little difference in original electrical properties. Their stability in water, however, is widely divergent. Dibenzo GMF-Pb₃O₄-sulfur cures are best, followed by the GMF-Pb₃O₄-Altax-no sulfur cures. GMF-Pb₃O₄-sulfur is good and GMF-Altax-Sulfur is slightly poorer. The Dibenzo GMF-Altax-Sulfur cures are quite poor, understandably, since it was observed that they were somewhat porous.

D. Dielectric Strength

Tests were conducted according to the short time method, ASTM D149-44, on cured pads prepared by scrubbing with acetone and drying thoroughly. It is well known that dispersion of compounding ingredients exerts a strong influence on dielectric strength and that straining the compound helps reduce the gross inhomogeneities. Thus it is understandable that changes in mixing technique could affect dielectric strength.

1. Mixing Technique

In past work, normally mixed standard formulations gave dielectric strengths varying between 300 and 550 volts per mil on 0.020" thick specimens. By employing the special mix (Tables VII, IX, XVI) the strength can be increased to 550-700 volts per mil. A hot Banbury mix at 325-360°F. provides the same returns (Table XIV). Here also is shown the advantage in dielectric strength of Whitetex over natural whiting.

Cab-O-Sil (Table XII) in a normally mixed compound, appears to give advantage in strength. This may be due in part to the heat generated in mixing such a stiff stock.

2. Effect of Unsaturation

Increasing unsaturation and molecular weight (Table XVI) slightly increase dielectric strength over GR-I-35. GR-I-17 appears exceptional. It should be stated that over the cure times employed in this work dielectric strength is fairly constant. The addition of Kenflex A is somewhat deleterious.

3. Affect of Cure Type

The quinoid type cure can be varied considerably as shown in Table VIII with resultant effects on the dielectric strength. It is apparent here that the best strength is obtainable from a GMF-Pb₃O₄-sulfur and a GMF-Pb₃O₄-Altax type of cure. In the evaluation of unsaturation

effects (Table XVI), Kenflex A appeared deleterious; however, in this work it is, without exception, beneficial. This is not an anomaly and can be explained readily. The control stocks, without Kenflex A, in Table VIII were all given a normal mix. All other stocks in both series were given the special hot mix. This once again points up the value to dielectric strength of hot mixing.

4. Effect of Polyac Treat

The Polyac treat (Table VII) is effective in increasing dielectric strength. Since both stocks had the benefit of a hot mix, it follows that the Polyac treat is alone responsible.

5. Effect of Heat Aging

One day of heat aging at 300°F. of the standard formula (Table IX) does not adversely affect dielectric strength. Three days at 300°F., however, produces degradation.

6. Effect of Carbon Black and Neoprene

The addition of 10 parts of SRF black (Table XVII) to an insulation stock reinforces the physical properties, but has no effect on the press cured dielectric strength. In this experiment there is an inconclusively small effect on dielectric strength of steam pressure cures. The dielectric strength of steam cures, however, is lower than press cures in both stocks.

Small amounts of Neoprene have been added to sulfur cures (Table XVIII) with advantageous effects (550 to 720 volts per mil) on dielectric strength. This advantage must be balanced by poorer dielectric constant, power factor, a 20-30 per cent loss in tensile strength, and a 5 per cent loss in elongation.

IV Properties of Sulfur Cures

A. Acceleration

Accelerator studies are generally run at temperatures ranging between 280°F. and 330°F. This necessitates an extrapolation of results to the high temperatures employed in continuous vulcanization. In order to give a better picture of the vulcanizate behavior at high temperatures, the present study was run at 400°F. At this temperature, cures are accomplished in terms of seconds rather than minutes. Charging stock and removal of press cures rapidly, posed a problem which was solved by use of a Zapp modified Mooney Viscosimeter. This had been converted to the purpose by replacing the stators with platens. Manual operation of such a press permitted a series of cures ranging from 5 to 120 seconds to be made with an error of less than 3 seconds. Silicon oil was used as a mold lubricant and cures were ice water quenched. The base formula employed was as follows:

| | |
|--------------|------------------------|
| GR-I-15 | 100 |
| Zinc Oxide | 5 (except where noted) |
| Stearic Acid | 1 |
| Whitetex #2 | 135 |
| Sulfur | 2 (except where noted) |
| Accelerators | As noted |

Data are shown graphically in Figures 3 and 4. Mooney scorch at 250°F. is also shown in the graph of each accelerator combination. The first figure represents the minutes to a viscosity increase of one point over the low; the second figure, minutes to a viscosity increase of one point per minute.

Accelerators are employed on the basis of molecular equivalent weights to either one or two parts of Tetramethylthiuram disulfide and Mercapto benzo thiazole always at 0.5 part.

Tellurac, Cumate, and Bismate all give fast cure induction, the latter two with low scorching tendency. Tetrone A, although not as fast in induction, exhibits a fast rate of cure and attains an exceptional state of cure. It is not scorchy. This is particularly interesting since at conventional temperatures Tetrone A is not unusual. The addition of M.B.T. as a secondary accelerator in all cases speeds the cure rate, increases the cure state, and promotes overcuring with respect to tensile strength. The combination of Tetrone A, Cumate and M.B.T. yields a good balance of fast induction, fast rate, reasonably high cure state, and a minimized tendency to overcure with respect to tensile. An increase in zinc oxide to 10 parts and sulfur to 3 parts further enhances these attributes without increasing scorch. It is felt that the high zinc oxide is primarily responsible for this effect.

A GMF Altax stock is included for comparison. This type of cure provides an extremely high state of cure, but is slower in induction and in rate of cure and has lower tensile strength.

B. Miscellaneous

Heat aging of Butyl-thiuram-sulfur cures is good compared to other rubbers but is quite inferior compared to the quinoid cure. This

is typified by the heat aging of a stock shown in Table XII. After 2 days at 300°F. in an air oven, only 10 per cent of the tensile strength is retained in a Tellurac-B.J.F. cure, while 48 per cent is retained in a Dibenzo GMP cure.

Ozone resistance of the sulfur cures is comparable to that of similarly loaded quinoid cures above the level of GR-I-35 unsaturation. However, the resistance of a quinoid cured GR-I-35 is greatly in excess of that of the same Butyl with a sulfur cure. Kenflex A in sulfur cures is effective in improving ozone resistance but yields a lesser improvement than in quinoid cures.

Electrical stability in water (Table XII) is strikingly better for quinoid cures. In this instance, it is felt that the magnitude of difference is due in part to the nature of the filler Cab-O-Sil. In Table XIX where a low whiting load was used, the sulfur cure shows better stability in dielectric constant although the quinoid cure shows markedly better stability in power factor.

V Jacketing

Butyl is inherently not adapted for applications where oil and flame resistance are imperative. There are, however, many applications where these requirements are not necessary and good physical properties, resistance to wear and weather are sufficient.

The development of Butyl carbon black stocks with greatly improved physical properties, resilience, and wearing qualities has been achieved recently, (5), (6), (7), (8). Gessler has found that this is accomplished best by dynamic heat treatment in a Banbury at temperatures in excess of 400°F., and serves to disperse and chemically cross link the carbon black with the polymer. The usual first criticism of such a process is that the Banbury will not continually stand such heat. Both Banbury manufacturers and field experience hold that no harm is done as long as the bearings are kept cool. Channel blacks, by virtue of the chemisorbed oxygen on their surface, heat interact with Butyl adequately although the interaction can be speeded and improved by promoters.

The furnace and thermal blacks require a promoter such as 0.2 parts/100 polymer of sulfur or 0.25 to 0.5 parts of Polyac or GMF. Usually 15-20 minutes hot Banburying is sufficient.

The immediate advantage of heat treating is evident in improved processing and reduced porosity, Figure 5. These stocks were GR-I-35,

100 parts, and carbon black 60 parts. The first sample in each group was extruded from normally mill mixed stock. The second sample was from a heat treated stock, the MT, SRF and HMF blacks being promoted with 0.2 parts sulfur. The heat interaction is improved by increasing Butyl unsaturation; therefore, selection of GR-I, GR-I-15, or even GR-I-25 would yield valuable advantages.

The process was originally developed using 30 minute cycles of autoclave heating interspersed with 5 minute cold milling. Data in Table XX show that heat interaction disperses the black, breaks chain structure and provides a high order of electrical resistivity. This effect can be promoted chemically. Subsequent work has shown that a promoted 400°F.+ Banbury mix of 15-20 minutes will accomplish the same results as many static cycles. SRF blacks will respond in the same fashion if similarly promoted; however, the furnace blacks HAF, HMF and CF show little electrical response, due probably to their strong reticulate structure.

The heat interaction process yields improved abrasion resistance. Three types of carbons were mixed with both the normal and the heat treat procedures in GR-I-18 stocks containing 50 parts of black. Curatives included 5 parts ZnO, 2 parts of sulfur, and 1.25 parts tetramethyl thiuram disulfide. Abrasion loss as a function of time of vulcaniation is shown in Figure 6. Results were obtained on a Lambourn abrader.

The heat treatment also removes a great deal of the stiffness usually observed in Butyl carbon black stocks and imparts a remarkable flexibility (Figure 7). Although heat treated stocks do not shown an advantage in low temperature properties (Table XII) the gain in physical properties is sufficient to offset the loss incurred through the addition of low temperature plasticizers.

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TABLE I

PROPERTIES OF VARIOUS FILLERS

Base Formula - Normal Mix
CR-I 35 100
Stearic Acid 1
Filler (as indicated) 90

| Filler | Whitetex #2 | Burgess Iceberg | Purecal U | Supermultifex | Talc 549 | Dicalite SA-3 | Alon S | Zeolox 20 |
|--------------------|-------------|-----------------|--------------|---------------|--------------------|---------------|--------|-----------|
| Extrusion @ 220°F. | 1 1/2 Royle | 120 RPM | 0.1815" dia. | | | | | |
| Rate in./min. | 127 | 142 | 118 | 100 | 154 | 117 | 99 | |
| Swell g./in. | 1.26 | 1.21 | 1.13 | 1.60 | 0.74 | 0.85 | 0.63 | |
| Index cc./in. | 0.88 | 0.85 | 0.80 | 1.11 | V. Rough & Swollen | 0.55 | 0.56 | 0.46 |

Acceleration for Cure on above Base Formula MgO, 10, ZnO, 5, Dibenzoz GMP, 6, Pb₃O₄, 10, Sulfur, 1, Wax, 5.

Physical Properties

Tensile (psi)-Elongation (%)

Mod. @ 200% (psi)-Shore

30' Cure @ 287°F.

45' " " "

60' " " "

Electrical Properties @ 1 KC and 76°F. No electrode weights

Dielectric Constant - Power Factor (%)

3.09-0.38

3.14-0.38

3.16-0.87

3.09-0.44

2.94-0.32

3.59-1.35

3.09-0.42

4.19-11.0

Resistivity

ohm cm

10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴10¹³10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴10¹⁴

TABLE II

HOT BANBURY VS. COLD MILL MIX

| | | | | |
|---|------------|--------------|----------|---------|
| Formula | | | | |
| GR-I 35 | | 100 | | |
| Zinc Oxide | | 5 | | |
| Stearic Acid | | 2 | | |
| Sulfur | | 2 | | |
| GMP | | 2 | | |
| Benzothiazyl Disulfide | | 4 | | |
| Filler | | As indicated | | |
| Filler (pts/100 gum) | | | | |
| Natural Whiting | 135 | 90 | 65 | |
| Whitetex | | 45 | 70 | 135 |
| <u>Banbury Mixed Start Temp. 280°F. Dump Temp. 325 - 360°F.</u> | | | | |
| Extrusion @ 260°F. Barrel | 210°F. Die | 0.4" Die | #1 Royle | |
| Rate in/min. | 111 | 118.5 | 141.6 | 167.8 |
| Swell cc/in. | 3.63 | 3.34 | 3.31 | 2.48 |
| Physical Properties | | | | |
| Tensile (psi) Elongation % | | | | |
| Mod. @ 200% (psi) Shore | | | | |
| 30' cure at 287°F. | 530-650 | 710-640 | 810-670 | 550-590 |
| | 180- 52 | 240- 52 | 350- 57 | 360- 71 |
| 45' cure at 287°F. | 670-660 | 780-660 | 920-670 | 600-550 |
| | 220- 52 | 280- 53 | 350- 57 | 430- 70 |
| 60' cure at 287°F. | 650-660 | 590-590 | 820-660 | 630-550 |
| | 230- 52 | 270- 53 | 390- 59 | 440- 73 |
| <u>Normally Mixed - Mill at 160°F.</u> | | | | |
| Extrusion as above | | | | |
| Rate in/min. | 93 | 110 | 140 | 149.5 |
| Swell cc/in. | 3.43 | 3.70 | 3.58 | 2.80 |
| Physical Properties | | | | |
| 30' cure @ 287°F. | 450-720 | 580-700 | 560-630 | 590-580 |
| | 110- 48 | 170- 49 | 170- 50 | 330- 67 |
| 45' cure @ 287°F. | 670-790 | 640-710 | 780-710 | 580-540 |
| | 120- 48 | 170- 49 | 220- 51 | 370- 67 |
| 60' cure @ 287°F. | 480-650 | 610-650 | 800-690 | 550-460 |
| | 170- 49 | 200- 52 | 250- 52 | 410- 67 |

TABLE III

EFFECT OF PROCESS OIL ON OZONE RESISTANCE

| | | | |
|------------------------------|---|-----|-----|
| GR-I 50 | 100 | 100 | 100 |
| Zinc Oxide | 5 | 5 | 5 |
| Stearic Acid | 3 | 3 | 3 |
| Sulfur | 2 | 2 | 2 |
| Clay | 100 | 100 | 100 |
| Tetramethylthuriam Disulfide | 1 | 1 | 1 |
| Process Oil | | 5 | 15 |
| Ozone Resist. | Cured 20 minutes at 307°F. 0.075 inch thick | | |
| T- 50 Dumbell | | | |
| 100% Elongation | | | |
| 0.012% O ₃ | | | |
| Mins. to Break | 95 | 35 | 20 |

TABLE IV

EFFECT OF RESINS AND POLYMERS ON OZONE RESISTANCE OF GR-I 35 VULCANIZATES

| Standard Formula | | |
|--------------------------------|--------------|--|
| GR-I 35 | 100 | |
| Zinc Oxide | 5 | |
| Stearic Acid | 1.5 | |
| Whitetex #2 | 90 | |
| Sulfur | 1.5 | |
| Dibenzo GMP | 6 | |
| Pb ₃ O ₄ | 9 | |
| Polymer or Resin | As indicated | |

| | | |
|---------------------|---|---------------|
| Cure | 32 minutes at 320°F. - 0.018 inch thick | |
| Type of Test | Constant Strain | 50% 100% |
| Ozone Concentration | 0.2 per cent by volume | |

| Resin or Polymer | Parts by Wt. | Minutes to Break | |
|------------------------|--------------|------------------|-----|
| | Added | | |
| None | - | 334 | 79 |
| Kenflex A | 5 | 854 | 160 |
| " | 10 | 3145 | 230 |
| " | 20 | 3145+(No | 520 |
| | | crack.) | |
| Polystyrene | 10 | 384 | |
| Amberol ST137X | 10 | 254 | |
| Piccolyte S115 | 10 | 946 | |
| Piccolastic D75 | 10 | 250 | |
| Alcova [®] #7 | 10 | 284 | |

| | Parts by Wt. | |
|----------------|--------------|------|
| | for GR-I 35 | |
| Parapol S-60 | 10 | 550 |
| " | 20 | 660 |
| " | 30 | 367 |
| Vistanex B-100 | 10 | 774 |
| " | 20 | 1074 |
| " | 30 | 2350 |

TABLE V
EFFECT FILLER ON OZONE RESISTANCE

Base Formula

| | |
|--------------------------------|--------------|
| GR-I 50 | 100 |
| Zinc Oxide | 5 |
| Stearic Acid | 3 |
| Sulfur | 3 |
| Dibenzo GMF | 6 |
| Pb ₃ O ₄ | 10 |
| Softener | 10 |
| Filler | As indicated |

| Filler | Cure 5 minutes at 300°F. Modulus at 200% Elongation psi. | 0.075" thick pads Minutes Ozone Resistance | |
|--|--|---|----------------|
| | | 0.012% O ₃ | 50% Elongation |
| Diatomaceous Earth 48.5 } Natural Whiting 73.3 } | 140 | | 180 |
| Diatomaceous Earth 48.5 } Clay 73.3 } | 190 | | >300 |
| Diatomaceous Earth 48.5 } Hydrated Alumina 73.3 } | 180 | | 290 |

TABLE VI

EFFECT OF STATE OF CURE (QUINOID SYSTEM) ON OZONE RESISTANCE

Standard Formula

| | |
|--------------------------------|-----|
| Butyl | 100 |
| Zinc Oxide | 5 |
| Stearic Acid | 1.5 |
| Sulfur | 1.5 |
| Dibenzo GMF | 6 |
| Pb ₃ O ₄ | 9 |
| Whitetex | 90 |

| <u>Butyl Grade</u> | <u>Cure Time @ 320°F.</u> | <u>Modulus psi at 200% Elongation</u> | <u>Ozone Resistance Minutes to Break 0.2% Ozone 50% Elongation Pads 0.018 inch thick</u> |
|--------------------|---------------------------|---|--|
| GR-I 35 | 16 minutes | 330 | 439 |
| | 32 | 340 | 325 |
| | 64 | 390 | 324 |
| GR-I 50 | 16 | 370 | 150 |
| | 32 | 380 | 160 |
| | 64 | 430 | 152 |
| GR-I 15 | 16 | 360 | 46 |
| | 32 | 380 | 66 |
| | 64 | 410 | 58 |
| GR-I 17 | 16 | 400 | 60 |
| | 32 | 400 | 86 |
| | 64 | 440 | 150 |
| GR-I 25 | 16 | 380 | 22 |
| | 32 | 390 | 37 |
| | 64 | 450 | 49 |

TABLE VII

EFFECT OF POLYAC TREAT - OZONE RESISTANCE
DIELECTRIC STRENGTH

| Formula Polyac Treat Mix | Standard GR-I 35 Special | Standard 0.2 pts/100 gum (Hot milled with Polymer 5 minutes @ 300°F.) |
|---|---|--|
| Cures | Avg. of 16, 32, 64' cures @ 320°F. - .018" thick. | |
| Ozone Resistance Mins. to Break 0.2 per cent O ₃ 50 per cent elongation | 510 | 908 |
| Dielectric Strength Short Time Test Volts/Mil. | 593 | 687 |
| Tensile (psi) Elongation % Modulus @ 200% (psi) - Shore | | |
| 16 Min. Cure @ 320°F. | 910-660 290-52 | 940-660 300-53 |
| 32 Min. Cure @ 320°F. | 940-660 300-53 | 930-650 340-53 |
| 64 Min. Cure @ 320°F. | 900-640 340-55 | 960-630 380-55 |

TABLE IX

EFFECT OF HEAT AGING ON OZONE RESISTANCE AND DIELECTRIC STRENGTH

| Formula (Special Mix) | | |
|--|---|---|
| GR-I 35 | 100 | |
| Zinc Oxide | 5 | |
| Stearic Acid | 1.5 | |
| Whitetex #2 | 90 | |
| Sulfur | 1.5 | |
| Dibenzo GMF | 6 | |
| Pb ₃ O ₄ | 9 | |
| Cure | 32 Minutes at 320°F. | |
| Samples | .018 inch Thick Pads. | |
| <u>Hours Aged in</u> <u>Geer Oven at 300°F.</u> | <u>Minutes Ozone Resistance</u> <u>0.2% O₃ 50% Elongation</u> | <u>Dielectric Strength</u> <u>Volts/Mil. Short Time Test</u> |
| Unaged | 440 | 606 |
| Aged 1/2 Hour | 595 | 637 |
| 8 Hours | 961 | 542 |
| 24 " | 848 | 684 |
| 72 " | (2550) No Cracking | 398 |

TABLE X

EFFECT OF UNSATURATION AND KENFLEX A ON HEAT AGING

GR-I-35 Standard Formula - Special Mix
 Press Cure - 32 minutes at 320°F.
 Aged 3 days at 300°F. - Air Oven
 Resin 10 parts on the standard where used.

| Polymer | Unaged | | | Aged | | |
|---------------------|-------------|---------------------|---------------|-----------------------------------|---------------------|---------------|
| | Tensile-psi | Elongation-per cent | Mod. 200% psi | Tensile-psi | Elongation-per cent | Mod. 200% psi |
| | | | | Figure in () = per cent retained | | |
| GR-I-35 | 880 | 620 | 340 | 370(42) | 420(68) | 280(82) |
| GR-I-35 + Kenflex A | 580 | 570 | 350 | 330(57) | 430(75) | 290(83) |
| GR-I-50 | 730 | 570 | 380 | 330(45) | 310(54) | 270(77) |
| GR-I-50 + Kenflex A | 510 | 520 | 370 | 340(67) | 330(63) | 290(88) |
| GR-I-15 | 580 | 520 | 380 | 270(47) | 270(52) | 250(66) |
| GR-I-15 + Kenflex A | 550 | 490 | 400 | 360(66) | 310(63) | 320(80) |
| GR-I-17 | 750 | 570 | 400 | 260(35) | 300(53) | 240(60) |
| GR-I-17 + Kenflex A | 670 | 540 | 420 | 340(51) | 300(56) | 310(74) |
| GR-I-18 | 750 | 560 | 400 | 350(47) | 320(57) | 300(75) |
| GR-I-18 + Kenflex A | 740 | 540 | 440 | 370(50) | 340(63) | 350(80) |
| GR-I-25 | 630 | 510 | 390 | 290(46) | 300(59) | 260(67) |
| GR-I-25 + Kenflex A | 550 | 470 | 400 | 360(65) | 290(62) | 340(85) |

Table XI

Effect of Additives on Heat Aging

GR-I-35 Standard Formula - Normal Mix
 Press Cure 32 minutes at 320°F.
Aged 3 days at 300°F. - Air Oven

| Additive (parts) | <u>Unaged</u> | | <u>Aged</u> | |
|----------------------------------|----------------|------------------------|-----------------------------|------------------------|
| | Tensile psi | Elongation per cent | Tensile psi | Elongation per cent |
| | | | Figures in () = % Retained | |
| None | 630 | - 620 | 170 (27) | - 360 (58) |
| Kenflex A (5) | 530 | - 620 | 190 (38) | - 350 (57) |
| " (10) | 560 | - 600 | 210 (38) | - 350 (58) |
| " (20) | 530 | - 520 | 270 (51) | - 360 (69) |
| Kenflex A (10) | | | | |
| Cab-O-Sil (5) | 700 | - 610 | 320 (46) | - 280 (46) |
| Kenflex A (10) | | | | |
| Cab-O-Sil (10) | 830 | - 590 | 370 (45) | - 280 (47) |
| <u>Substituted for</u> | | | | |
| <u>Equal Wt. GR-I-35 (parts)</u> | | | | |
| Parapol S-60 (10) | 650 | - 570 | 200 (31) | - 330 (58) |
| " (20) | 640 | - 430 | 180 (28) | - 220 (51) |
| " (30) | 640 | - 330 | 200 (31) | - 110 (34) |
| Vistanex B100 (10) | 770 | - 610 | 200 (20) | - 340 (56) |
| " (20) | 670 | - 590 | 170 (26) | - 320 (54) |
| " (30) | 650 | - 570 | 160 (25) | - 300 (53) |

Table XII

Cab-O-Sil Stock

| Formula | - | Normal Mix | |
|--------------------------------|---|------------|-----|
| GR-I-15 | | 100 | 100 |
| Zinc Oxide | | 5 | 5 |
| Stearic Acid | | 2 | 2 |
| Sulfur | | 1.5 | 2 |
| Dibenzo GMF | | 6 | |
| Pb ₃ O ₄ | | 10 | |
| Aranox | | 0.5 | |
| Cab-O-Sil | | 49 | 49 |
| Tellurac | | | 1 |
| B.J.F. | | | 1 |

Press Cure 32 minutes at 320°F.

Original Physicals

| | | |
|------------------------------|----------|----------|
| Tensile (psi) Elongation (%) | 1770-620 | 2370-940 |
| Mod. @ 200%(psi) Shore | 460- 78 | 310- 78 |

Aged Air Oven 300°F.

Tensile - Elong. - Shore

Figures in () % Retained

| | | |
|--------|--------------------|----------------------|
| 2 days | 850(48)-280(45)-85 | 250(10.5)-540(58)-82 |
| 4 days | 630(36)-240(39)-85 | |

Water Absorption Mg/sq.in. at 85°C.

| | | |
|---------|------|------|
| 1 day | 4.5 | 21.7 |
| 14 days | 15.9 | 78.2 |

Electrical Properties @ 1KC after water immersion at 85°C.* - 6 Kg. wt.

Dielectric Constant - % Power Factor

| | | |
|----------|-------------|-------------|
| Original | 2.86 - 0.31 | 2.77 - 0.70 |
| 1 day | 2.98 - 1.5 | 4.32 - 13.5 |
| 7 days | 3.18 - 2.3 | - - |
| 14 days | 3.29 - 2.2 | - - |

Dielectric Strength volts/mil - short time test on 0.020 inch pads

692

817

* Determined at room temperature.

Table XIII

Heat Aging In Nitrogen

| Formula | - | Normal Mix |
|--------------------------------|---|------------|
| GR-I-50 | | 100 |
| Zinc Oxide | | 5 |
| Stearic Acid | | 3 |
| Sulfur | | 1.5 |
| Dibenzo GMF | | 6 |
| Pb ₃ O ₄ | | 10 |
| Clay | | 50 |

Press Cured 30 minutes at 287°F.

Unaged

Tensile (psi) - Elongation (%) - Shore 700-580-52

Aged Air Oven at 300°F.

Tensile - Elongation - Retained (%)

2 days 46 - 88

4 days 25 - 74

Aged Nitrogen at 300°F.

Tensile - Elongation - Retained (%)

5 days 51 - 96

10 days 44 - 80

15 days 32 - 87

20 days 16 - 90

Table XIV

Electrical Stability in Water & Dielectric Strength

Natural Whiting Vs. Whitetex

| | |
|------------------------|--------------|
| Base Formula | |
| GR-I-35 | 100 |
| Zinc Oxide | 5 |
| Stearic Acid | 2 |
| Sulfur | 2 |
| GMF | 2 |
| Benzothiazyl Disulfide | 4 |
| Filler | As Indicated |

Cured 30 minutes at 287°F.

Banbury mixed Start. Temp. 280°F. Dump 325°F. to 360°F.

| Filler (parts/100 gum) | Dielectric Strength volts/mil - pads 0.030 inch thick | Electrical Properties at 1 KC - No Wt. | |
|---------------------------------------|---|--|---|
| | | Original | After Water Immersion 7 days at room temp. |
| | | Dielectric Constant - % Power Factor | |
| Natural Whiting (135) | 520 | 3.26 - 0.27 | 3.33 - 0.67 |
| Natural Whiting (90) Whitetex (45) | 585 | 3.08 - 0.31 | 3.19 - 0.65 |
| Natural Whiting (65) Whitetex (70) | 585 | 2.99 - 0.28 | 3.04 - 0.54 |
| Whitetex (135) | 680 | 2.75 - 0.36 | 2.88 - 0.73 |
| <u>Normal Mill Mixed</u> | | | |
| Natural Whiting (135) | 485 | 3.14 - 0.36 | 3.17 - 0.70 |
| Natural Whiting (90) Whitetex (45) | 505 | 3.07 - 0.36 | 3.17 - 0.59 |
| Natural Whiting (65) Whitetex (70) | 520 | 3.01 - 0.38 | 3.05 - 0.57 |
| Whitetex (135) | 570 | 2.87 - 0.33 | 2.80 - 0.89 |

Table XV

Mechanical Water Absorption and Electrical Stability

In Water

GR-I-35 Standard Formula - Normal Mix
 Cure 32 minutes at 320°F. - 0.075 inch thick pads
 Additives as indicated

| Additive (parts/100 gum) | Mechanical Water Absorption Mg/sq. in. at 85°C. | | | Electrical Properties at 1 KC* After Water Immersion at 85°C. | | | |
|--|--|--------|---------|--|--------|--------|---------|
| | 1 day | 7 days | 14 days | Original | 1 day | 7 days | 14 days |
| | | | | Dielectric Constant at R.T. | | | |
| | | | | % Power Factor at R.T. | | | |
| None | 2.8 | - 8.5 | - 12.1 | 3.27 | - 3.38 | - 3.49 | - 3.50 |
| | | | | 0.64 | 0.68 | 0.78 | 0.85 |
| Kenflex A (5) | 2.9 | - 8.5 | - 11.9 | 3.19 | - 3.41 | - 3.48 | - 3.48 |
| | | | | 0.71 | 0.82 | 0.82 | 0.89 |
| Kenflex A (10) | 3.0 | - 8.7 | - 12.0 | 3.27 | - 3.46 | - 3.49 | - 3.52 |
| | | | | 0.7 | 0.92 | 1.01 | 1.08 |
| Kenflex A (20) | 3.2 | - 8.8 | - 12.2 | 3.30 | - 3.46 | - 3.49 | - 3.54 |
| | | | | 0.62 | 0.88 | 1.07 | 1.15 |
| Kenflex A (10) Cab-O-Sil (5) | 3.0 | - 7.0 | - 9.8 | 3.32 | - 3.44 | - 3.49 | - 3.52 |
| | | | | 0.57 | 0.68 | 0.60 | 0.61 |
| Kenflex A (10) Cab-O-Sil (10) | 3.3 | - 6.2 | - 8.6 | 3.32 | - 3.46 | - 3.49 | - 3.50 |
| | | | | 0.57 | 0.65 | 1.02 | 0.59 |
| <u>Substituted for Equal Parts Gum</u> | | | | | | | |
| Vistanex B100 (10) | 3.0 | - 7.6 | - 11.3 | 3.25 | - 3.40 | - 3.45 | - 3.51 |
| | | | | 0.53 | 0.76 | 1.04 | 1.19 |
| Vistanex B100 (30) | 2.9 | - 7.0 | - 10.4 | 3.22 | - 3.40 | - 3.47 | - 3.48 |
| | | | | 0.54 | 0.80 | 1.01 | 1.21 |

* 6 Kg weight on electrode.

Table XVI

Effect Unsaturation on Dielectric Strength

Standard Formula - Special Mix
Resin - 10 parts/100 gum where indicated
Samples - 0.018 inch thick pads

| Polymer | Dielectric Strength (Short Time Test) Volts/mil - Average value of 16,32,64 minutes Cures at 320°F. |
|---------------------|---|
| GR-I-35 | 693 |
| GR-I-35 + Kenflex A | 588 |
| GR-I-50 | 762 |
| GR-I-50 + Kenflex A | 432 |
| GR-I-15 | 640 |
| GR-I-15 + Kenflex A | 695 |
| GR-I-17 | 847 |
| GR-I-17 + Kenflex A | 831 |
| GR-I-18 | 763 |
| GR-I-18 + Kenflex A | 691 |
| GR-I-25 | 761 |
| GR-I-25 + Kenflex A | 661 |

Table XVII

Dielectric Strength

Press Vs. Steam Cures and Effect of Carbon Black

| Formula (Normal Mix) | <u>1</u> | <u>2</u> |
|--------------------------------|----------|----------|
| GR-I-35 | 100 | 100 |
| Zinc Oxide | 5 | 5 |
| Stearic Acid | 1 | 1 |
| Wax 130°F. | 5 | 5 |
| Sulfur | 1 | 1 |
| Dibenzo GMF | 6 | 6 |
| Pb ₃ O ₄ | 10 | 10 |
| Whitetex #2 | 90 | 90 |
| SRF Black | | 10 |

Press Cured 45 minutes at 287°F.

Tensile (psi) Elongation (%)

Mod. at 200% (psi) - Shore A

730-590
320- 58

800-580
350- 59

Dielectric Strength (short time test) 0.035 inch thick pads
volts/mil 543 548

Dielectric Strength - calendered stock cured 45 minutes at 40# steam

Shore Hardness 54 57
volts/mil 436 496

Table XVIII

Electrical Effects of Using Neoprene

| Formula | <u>1</u> | <u>2</u> | <u>3</u> | <u>4</u> |
|----------------------------------|---|----------|----------|----------|
| GR-I-50 | 100 | 90 | 100 | 90 |
| Neoprene GN | | 10 | | 10 |
| Zinc Oxide | 5 | 3 | 5 | 5 |
| Extra Lt. MgO | | 2 | | 4 |
| Stearic Acid | 3 | 3 | 1 | 1 |
| Tetramethyl Thiuram Disulfide | 1 | 1 | 1 | 1 |
| Mercaptobenzothiazole | 0.5 | 0.5 | | |
| Sulfur | 2 | 2 | 1.5 | 1.5 |
| Mineral Rubber | | | 20 | 20 |
| Diatomaceous Earth | | | 125 | 125 |
| Clay | 100 | 100 | | |
| Cure | 60 minutes @ 292°F. 25 minutes @ 287°F. | | | |
| Electrical Properties 1KC | | | | |
| Dielectric Constant | 2.54 | 3.03 | | |
| % Power Factor | 0.52 | 0.90 | | |
| Dielectric Strength volts/mil | | | 550 | 720 |

Table XIX

Electrical Stability

Sulfur Vs. Quinoid Cures

| | | |
|--------------------------------|-----|-----|
| GR-I-35 | 100 | 100 |
| Zinc Oxide | 5 | 5 |
| Sulfur | 2 | 1.5 |
| Tetramethyl Thiuram Disulfide | 1 | |
| Tellurac | 1 | |
| Wax | 5 | 5 |
| Stearic Acid | 2 | 2 |
| Dibenzo GMF | | 6 |
| Pb ₃ O ₄ | | 10 |
| Natural Whiting | 50 | 50 |

Physical Properties

Tensile (psi) - Elongation (%)

Mod. @ 200% (psi) - Shore

| | | | |
|-----------|----------|--------------|---------|
| 30' @ 307 | 1920-770 | 30' @ 287°F. | 640-660 |
| | - 35 | | 80- 43 |
| 60' " | 1870-750 | 60' " | 660-700 |
| | - 37 | | 100- 43 |
| 90' " | 1640-700 | 80' " | 750-680 |
| | - 38 | | 120- 42 |

Electrical Properties @ 1KC After water immersion at 70°C. - No weight

Dielectric Constant - % Power Factor 60' cures

| | | |
|----------|-------------|-------------|
| Original | 2.61 - 0.55 | 2.80 - 0.44 |
| 7 days | 2.77 - 3.3 | 3.16 - 0.52 |

Table XX

Electrical Resistivities of Butyl - Channel Black Compounds

Heat Treated With and Without Promoting Agents

| Promoting Agent No. of Cycles Heat Treatment | None | Sulfur | Polyac |
|--|-----------------------|-----------------------|-----------------------|
| 0 | 4.12×10^7 | 3.90×10^7 | 6.71×10^{10} |
| 1 | -- | 3.69×10^7 | 7.46×10^{14} |
| 2 | 5.58×10^7 | 1.96×10^9 | 10^{15} |
| 3 | -- | 3.41×10^{13} | 10^{15} |
| 5 | -- | 1.27×10^{14} | 10^{15} |
| 6 | 9.32×10^{10} | -- | -- |
| 8 | 3.22×10^{13} | 10^{15} | 10^{15} |
| 12 | 3.22×10^{14} | 10^{15} | 10^{15} |

Table XXI

Jacket Stocks

| | |
|---|---|
| Heat Treated Masterbatch | 00 Banbury Mix |
| GR-I-25 | Preheat Banbury |
| Philblack A | 0' Charge Butyl |
| Stearic Acid | 2' Charge 1/2 Black, Stearic Acid, Polyac |
| Polyac | 4' Charge 1/2 Black |
| | 9' Temp., 400°F. |
| | 17' Temp., 450°F. (shot cold water) |
| | 20' Dump Temp., 420°F. |
| Mill Mixes | |
| Masterbatch | |
| Fresh GR-I-25 | |
| Philblack A | |
| Zinc Oxide | |
| Stearic Acid | |
| Sulfur | |
| Tetramethyl Thiuram Disulfide | |
| Benzothiazyl Disulfide | |
| Process Oil | |
| Trioctyl Phosphate | |
| Tensile (psi) - Elongation (%) | |
| Mod. @ 200 % (psi) - Shore | |
| 20' cure 320°F. | |
| Tear Resistance Crescent B #/in. | |
| Low Temperature Tests @ -65°F. 2 hours and 7 days | |
| Gehman Torsional | |
| Stiffness Ratio | |
| Cold Tension Recovery (%) | |
| Brittleness Test | |

| | | | |
|----------|----------|----------|--------------|
| 1 | 2 | 3 | 4 |
| 300 | 360 | 360 | 360 |
| 120 | 60 | 60 | 60 |
| 15 | 15 | 15 | 15 |
| 1.5 | | | |
| 6 | 6 | 6 | 6 |
| 3 | 3 | 3 | 3 |
| 3 | 3 | 3 | 3 |
| | 30 | | 30 |
| 1650-350 | 2050-320 | 1690-340 | 1780-360 |
| 920- 64 | 1200- 60 | 870- 53 | 860- 53 |
| 96 | 90 | 75 | 70 |
| 25.6 | 31.5 | 9.65 | 6.9-7.57 (1) |
| 30 | 30 | 50 | 75-33 (1) |
| Failed | Failed | Failed | Passed - (2) |

- (1) Average of three samples
(2) Two failures - one pass

Table XXII

Compounding Ingredient

| | |
|---------------------------|--|
| Alon S | Godfrey L. Cabot, In., Boston, Mass. |
| Aranox | Naugatuck Chemical Division |
| Burgess Iceberg | Burgess Pigment Co., Sandersville, Ga. |
| Dibenzo GMF | Naugatuck Chemical Division |
| Dicalite SA-3 | The Dicalite Co., Chicago, Ill. |
| GMF | Naugatuck Chemical Division |
| Kenflex A | Kenrich Corp., Maspeth, N. Y. |
| Parapol S | Enjay Company, Inc., New York |
| Vistanex B100 | Enjay Company, Inc., New York |
| Purecal U | Wyandotte Chem. Corp., Wyandotte, Mich. |
| Supermultifex | Diamond Alkali Co., Pittsburgh, Pa. |
| Talc 549 | Eastern Magnesia Talc Co., Burlington, Vt. |
| Tributoxy ethyl phosphate | Ohio Apex, Inc., Nitro, W. Va. |
| Whitetex | Southern Clays, Inc., New York |
| Zeolex 20 | S. M. Huber Corp., New York |

FIGURE 1
EXTENSION APPARATUS

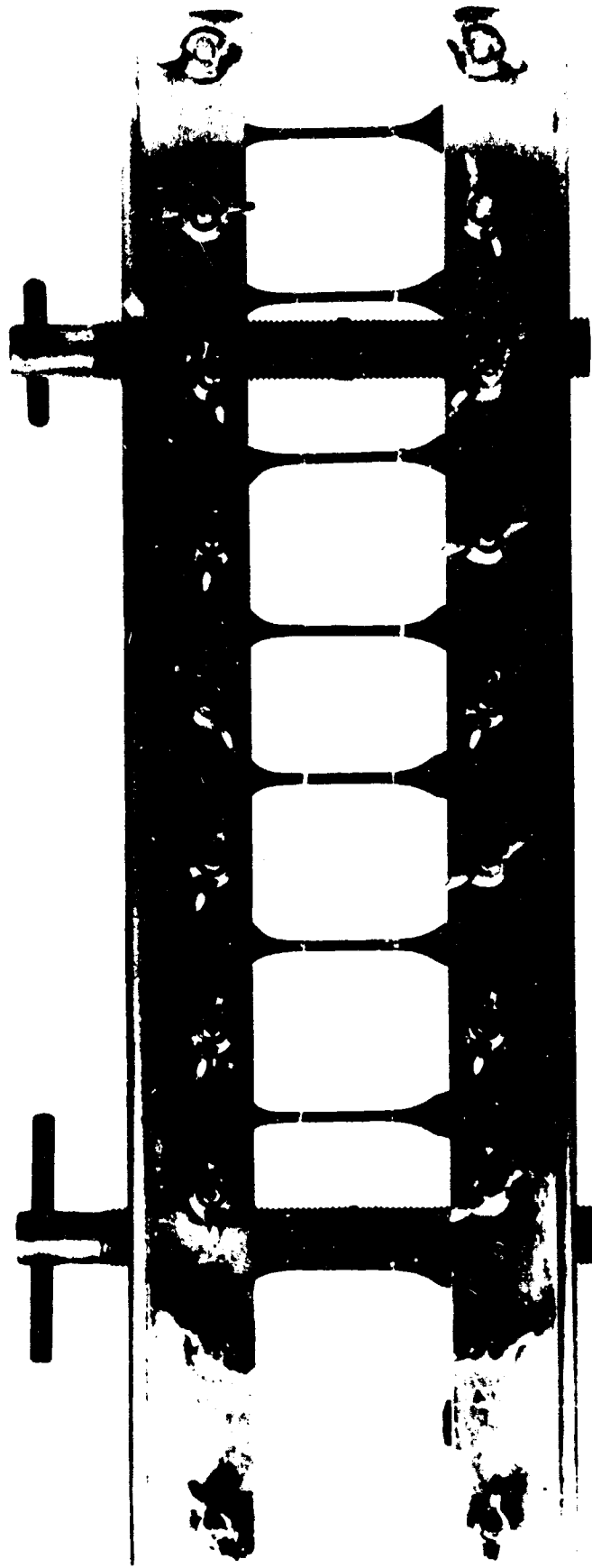


FIGURE 2
EFFECT UNSATURATION ON
OZONE RESISTANCE

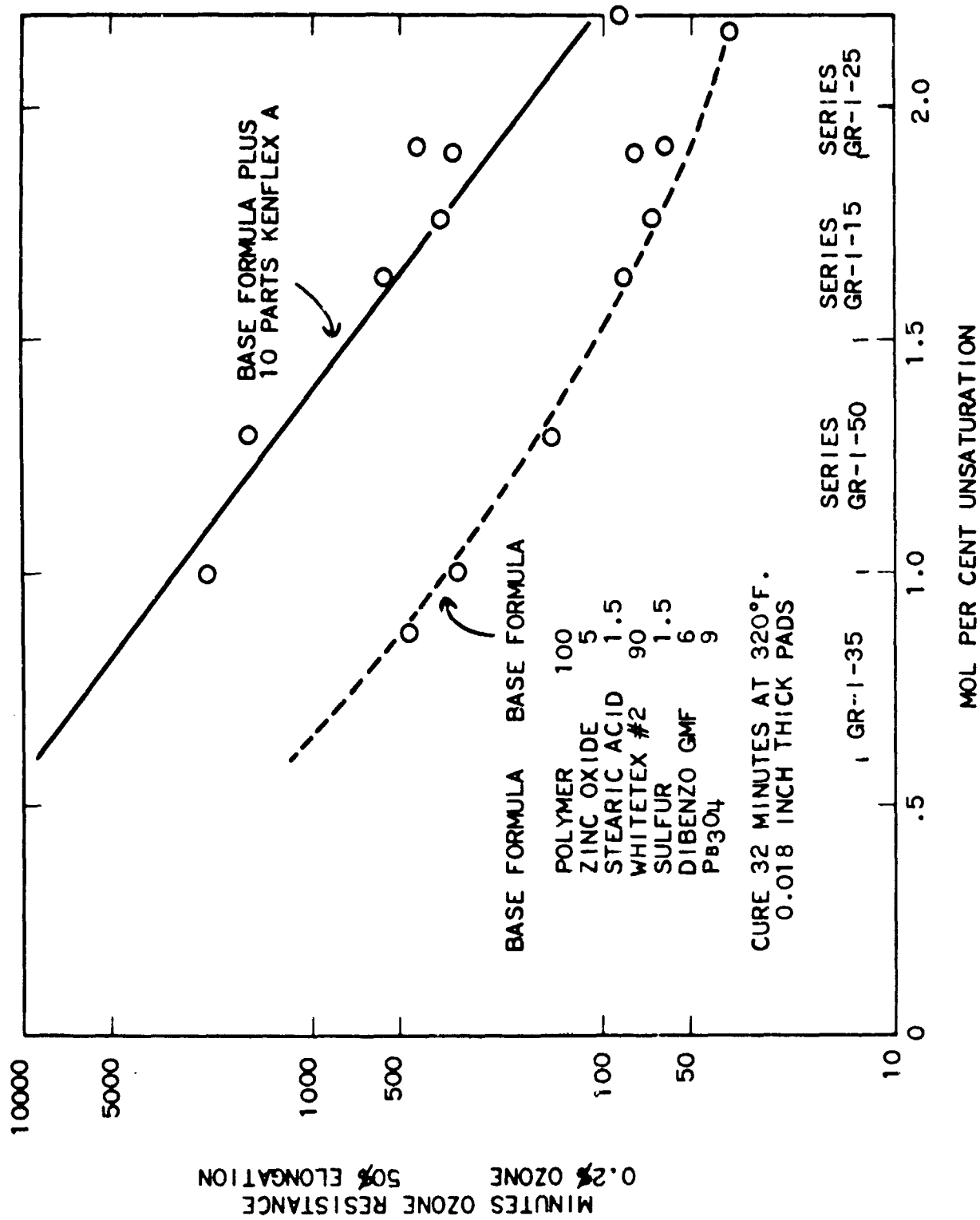


FIGURE 3
HIGH TEMPERATURE ACCELERATOR STUDY

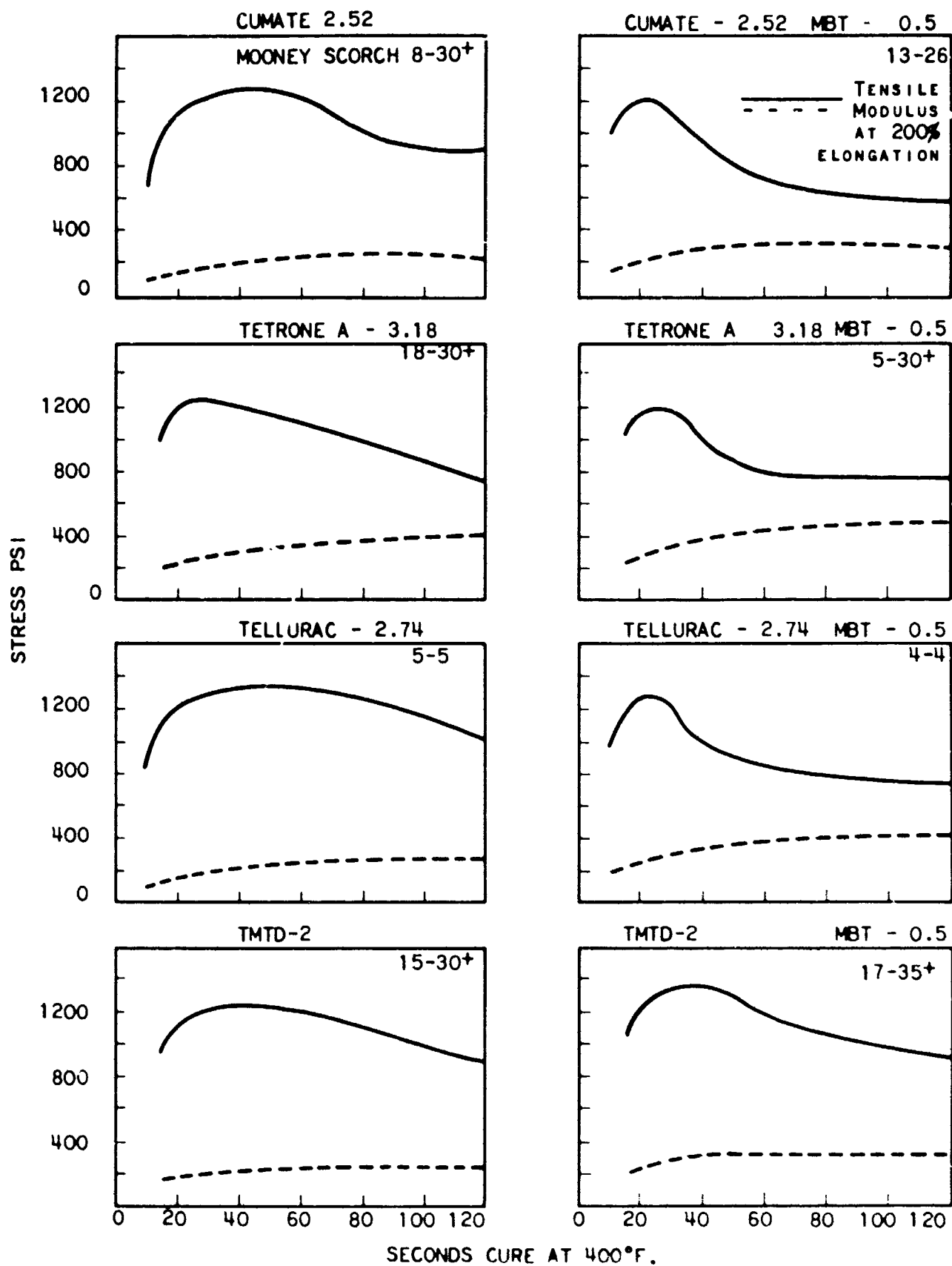
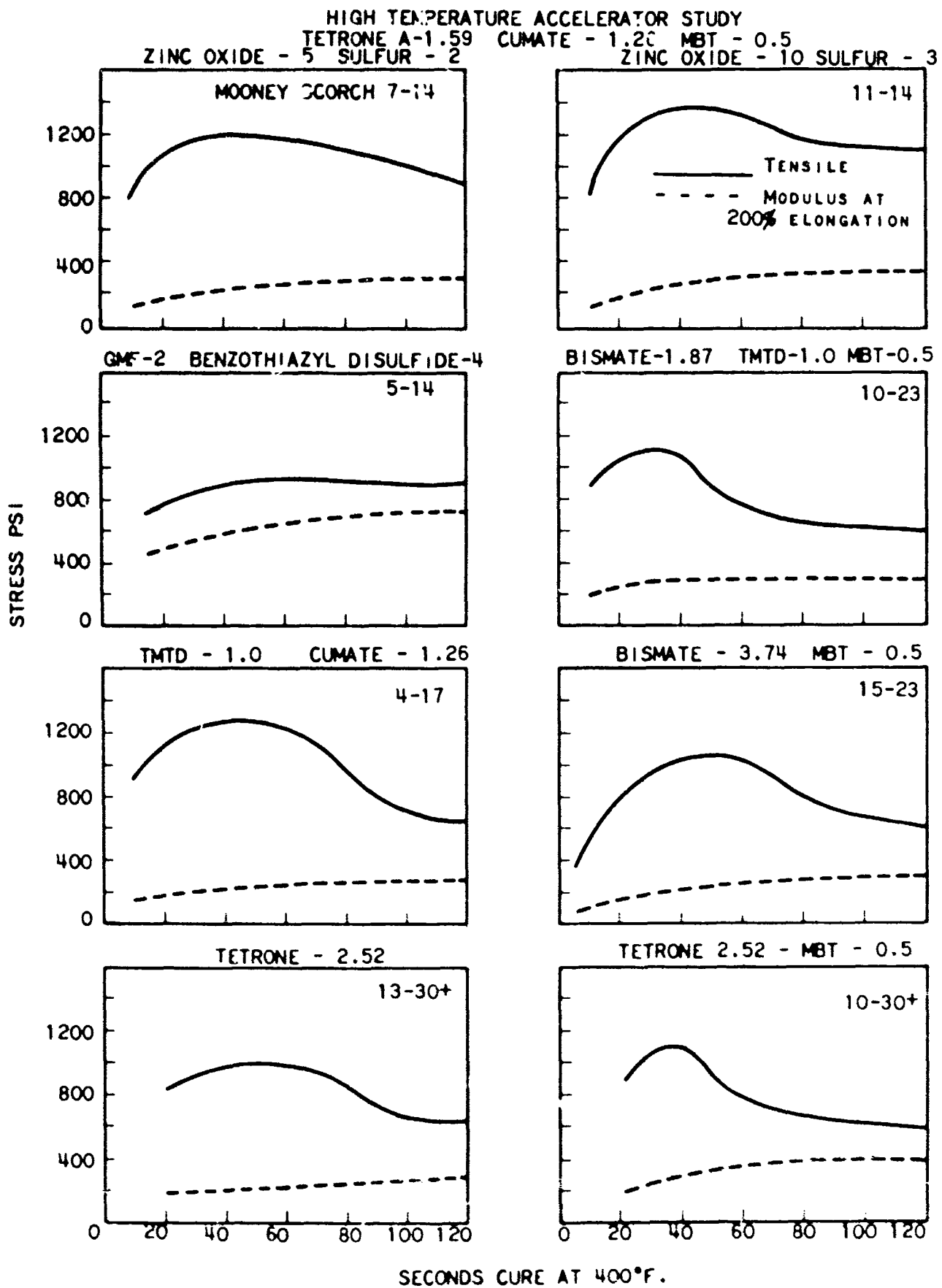


FIGURE 4



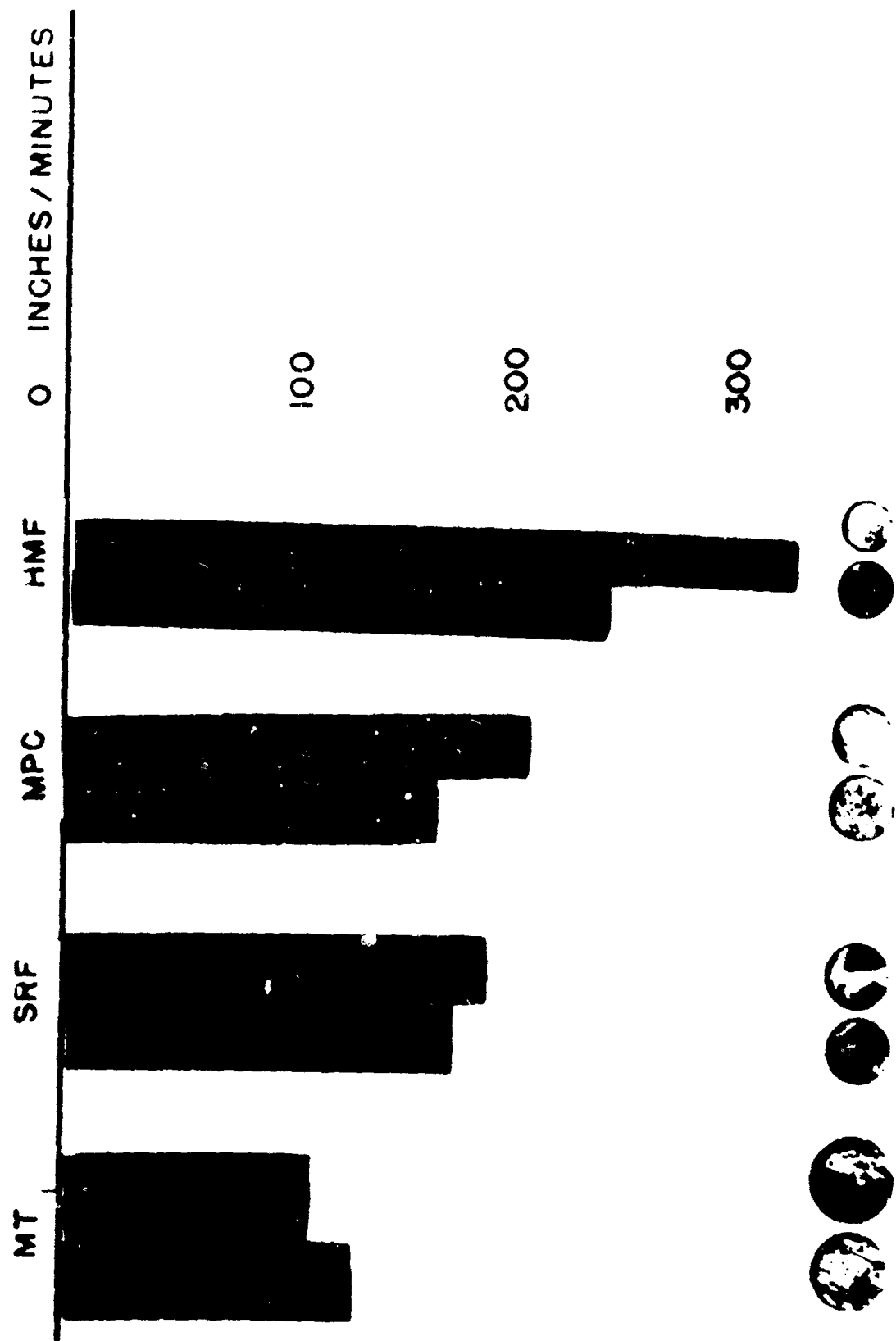


FIGURE 5
EFFECT OF HEAT INTERACTION
ON EXTRUSION

FIGURE 6

ABRASION RESISTANCE AND THE TYPE AND TREATMENT OF
CARBON BLACK MIXTURES

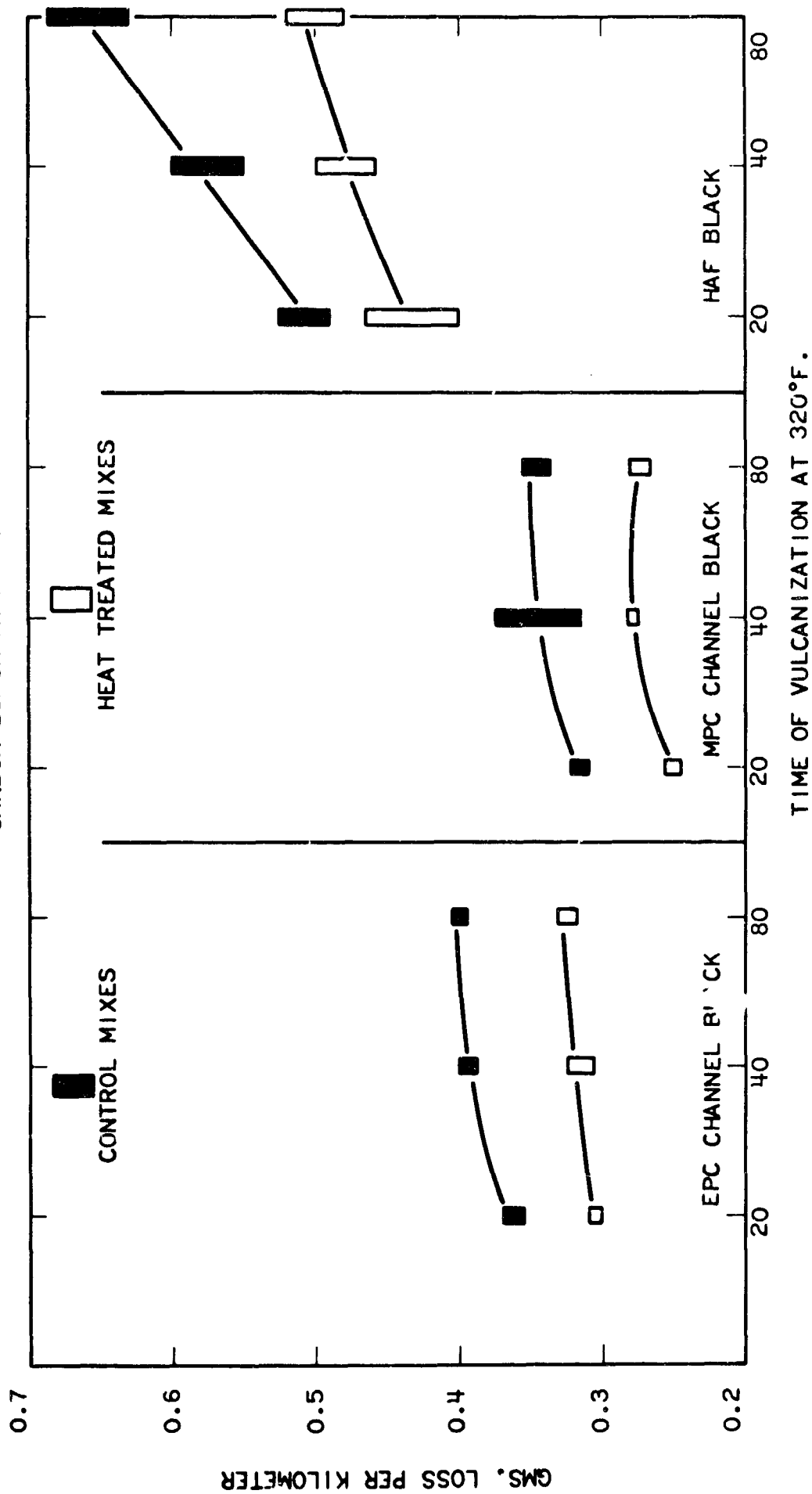


FIGURE 7

